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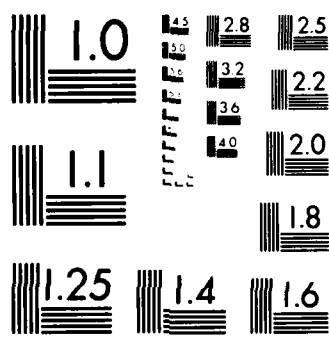
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THIN FILMS PROPERTIES OF SPUTTERED NIOBIUM SILICIDE

ON  $\text{SiO}_2$ ,  $\text{Si}_3\text{N}_4$  and on N+ Poly-Si

by

T. P. Chow\*, W.-J. Lu, A. J. Steckl, B. J. Baliga\*

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NIOBIUM SILICIDE

ON  $\text{SiO}_2$ ,  $\text{Si}_3\text{N}_4$  AND ON  $\text{N}^+$  POLY-SI

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ABSTRACT

The thin film properties of sputtered niobium silicide on  $\text{SiO}_2$ ,  $\text{Si}_3\text{N}_4$  and  $n^+$  doped poly-Si have been investigated. The structural and compositional properties were studied with X-ray diffraction, Rutherford backscattering spectrometry and secondary ion mass spectrometry.

## I. INTRODUCTION

Refractory metal silicides have become a key part of VLSI silicon device technology as MOS gates and interconnects due to their low resistivity, oxidation resistance and compatibility with MOS processes [1,2]. Among the silicides, the most widely used ones are  $\text{MoSi}_2$ ,  $\text{TaSi}_2$ ,  $\text{WSi}_2$  and  $\text{TiSi}_2$ . Recently,  $\text{NbSi}_2$  was also reported [3] to have similar properties as the other refractory silicides. In that study, slightly metal-rich ( $\text{Si/Nb} \sim 1.8$ ) silicide films obtained from a hot-pressed composite target in rf sputtering system were investigated. Those films were shown to contain  $\text{NbSi}_2$  as well as a significant amount of hexagonal  $\text{Nb}_5\text{Si}_3$ . Also, a resistivity of  $\sim 100 \mu\Omega\text{-cm}$  was measured after annealing at elevated temperatures.

In this paper, we report on improved thin film properties of niobium silicide on  $\text{SiO}_2$  and on  $n^+$  poly-Si.

## II. EXPERIMENTAL PROCEDURE

Silicon-rich ( $\text{Si/Nb} \sim 2.3$ ) silicide films were deposited at room temperature from a cold-pressed composite target (99.6%) in a dc magnetron sputtering system (Varian 3140). Rutherford Backscattering Spectrometry (RBS) was done with a 2 MeV  $^4\text{He}^+$  beam from a linear accelerator, Secondary Ion Mass Spectrometry with a Cameca IMS 3-f ion microscope and X-ray diffraction with a Siemens D-500 automated diffractometer.

## III. RESULTS

### (a) Structure and Composition -

The as-deposited films were essentially amorphous but after annealing at elevated temperatures, they exhibited a predominantly  $\text{NbSi}_2$  structure. Fig. 1a and b show X-ray diffraction patterns for an as-sputtered film and the same film after annealing in hydrogen at  $1000^\circ\text{C}$  for 60 min. The major niobium disilicide peaks are at  $2\theta$  of 21.4, 25.4, 40.1, 41.2 and  $47.0^\circ$ , corresponding to the (100), (101), (111), (003) and (112) planes respectively. Trace amounts of carbon-stabilized, hexagonal  $\text{Nb}_5\text{Si}_3$  components were also detected at 27.2, 29.2 and  $36.4^\circ$ . Pure  $\text{Nb}_5\text{Si}_3$  is stable in two tetragonal structures ( $\text{D8}_1$  (tI32) and  $\text{D8}_m$  (tI32)) while impurities, such as carbon, tends to stabilize it into the hexagonal structure ( $\text{D8}_g$ ) [4]. In fact, many of the other metal silicides ( $\text{Mo}_5\text{Si}_3$ ,  $\text{W}_5\text{Si}_3$ , and others) have the same characteristic and the detection of such a hexagonal silicide phase is indicative of the presence of carbon or other impurities.

#### (b) Resistivity -

The thin film resistivity of these  $\text{NbSi}_2$  films after annealing was observed to be consistently lower than the previously reported films. In Fig. 2, the sheet resistance of  $5600\text{\AA}$  niobium silicide on oxidized silicon substrates is shown as a function of annealing time at 800, 900 and  $1000^\circ\text{C}$  in hydrogen. As deposited, the films have a  $R_s$  of  $\sim 11 \Omega/\text{square}$ . The resistivity decreases rapidly within the first 15 min and changes only slightly for longer times. After annealing for 1 hr, the sheet resistance dropped to 2.8, 2.0 and  $1.3 \Omega/\text{square}$  for 800, 900 and  $1000^\circ\text{C}$  respectively. For the silicide film on  $\text{SiO}_2$ , the sheet resistance dropped from  $10.5 \Omega/\text{square}$  to  $1.4 \Omega/\text{square}$  ( $78 \mu\Omega\text{-cm}$ ) after 15 min and to  $1.3 \Omega/\text{square}$  ( $72 \mu\Omega\text{-cm}$ ) after 60 min. The lowest  $\text{NbSi}_2$  resistivity measured after annealing at  $1000^\circ\text{C}$  was  $\sim 70 \mu\Omega\text{-cm}$  which represents a  $\sim 30\%$  improvement over previous data on rf sputtered films and is



close to the value of  $\sim 50 \mu\Omega\text{-cm}$  reported for niobium on poly-Si films annealed under vacuum [1]. The bulk values, for comparison, range from 6.3 to  $50 \mu\Omega\text{-cm}$ , with the lower values probably erroneous [4]. Similar dependence on annealing time has been observed for niobium silicide on silicon nitride and on  $n^+$  poly-Si (polycide) and is shown in Fig. 3 and 4. For the polycide stack, the sheet resistance was decreased from 13.7 to  $2.8 \Omega/\text{square}$  after 30 min but increased to  $3.0 \Omega/\text{square}$  after 60 min. The rapid decrease in resistivity followed by a saturation-like characteristic when annealed at high temperatures ( $>700^\circ\text{C}$ ) has also been observed for other silicides, such as  $\text{TiSi}_2$  [5,6],  $\text{MoSi}_2$  [7,8] and  $\text{WSi}_2$  [8,9,10]. Also, when these niobium silicide films were annealed at different temperatures, similar dependence of sheet resistance on time was observed but higher saturation values were obtained for lower temperatures.

#### IV. SUMMARY

approx. microohms

Niobium silicide thin films deposited on  $\text{SiO}_2$ ,  $\text{Si}_3\text{N}_4$  and  $n^+$  poly-Si have been characterized. Similar to the other refractory silicides, annealing at high temperatures resulted in structural recrystallization and a sharp decrease in resistivity. After annealing at  $1000^\circ\text{C}$ , a resistivity of  $\sim 70 \mu\Omega\text{-cm}$  was obtained. For a  $2500\text{\AA} \text{ NbSi}_2 / 2500\text{\AA} n^+ \text{ poly-Si}$  stack, a sheet resistance of  $2.5 \Omega/\text{square}$  after annealing at the same temperature.

#### ACKNOWLEDGMENT

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Fig. 1 X-ray diffraction patterns of niobium silicide film on  $\text{SiO}_2/\text{Si}$  substrate: (a) as-sputtered and (b) after annealing at  $1000^\circ\text{C}$  for 30 min in  $\text{H}_2$ .

Fig. 2 Sheet resistance of  $5600\text{\AA}$ -thick  $\text{NbSi}_2$  on oxidized silicon substrate as a function of annealing time at various annealing temperatures ( $800\text{--}1000^\circ\text{C}$ ) in hydrogen.

Fig. 3 Sheet resistance of  $5600\text{\AA}$ -thick  $\text{NbSi}_2$  on  $\text{Si}_3\text{N}_4/\text{SiO}_2/\text{Si}$  substrate as a function of annealing time at various annealing temperatures ( $700\text{--}1000^\circ\text{C}$ ) in hydrogen.

Fig. 4 Sheet resistance of  $2500\text{\AA}$ -thick  $\text{NbSi}_2$  and  $2500\text{\AA}$ -thick  $n^+$  doped poly-Si on  $\text{SiO}_2/\text{Si}$  substrate as a function of annealing time at various annealing temperatures ( $700\text{--}1000^\circ\text{C}$ ) in hydrogen.

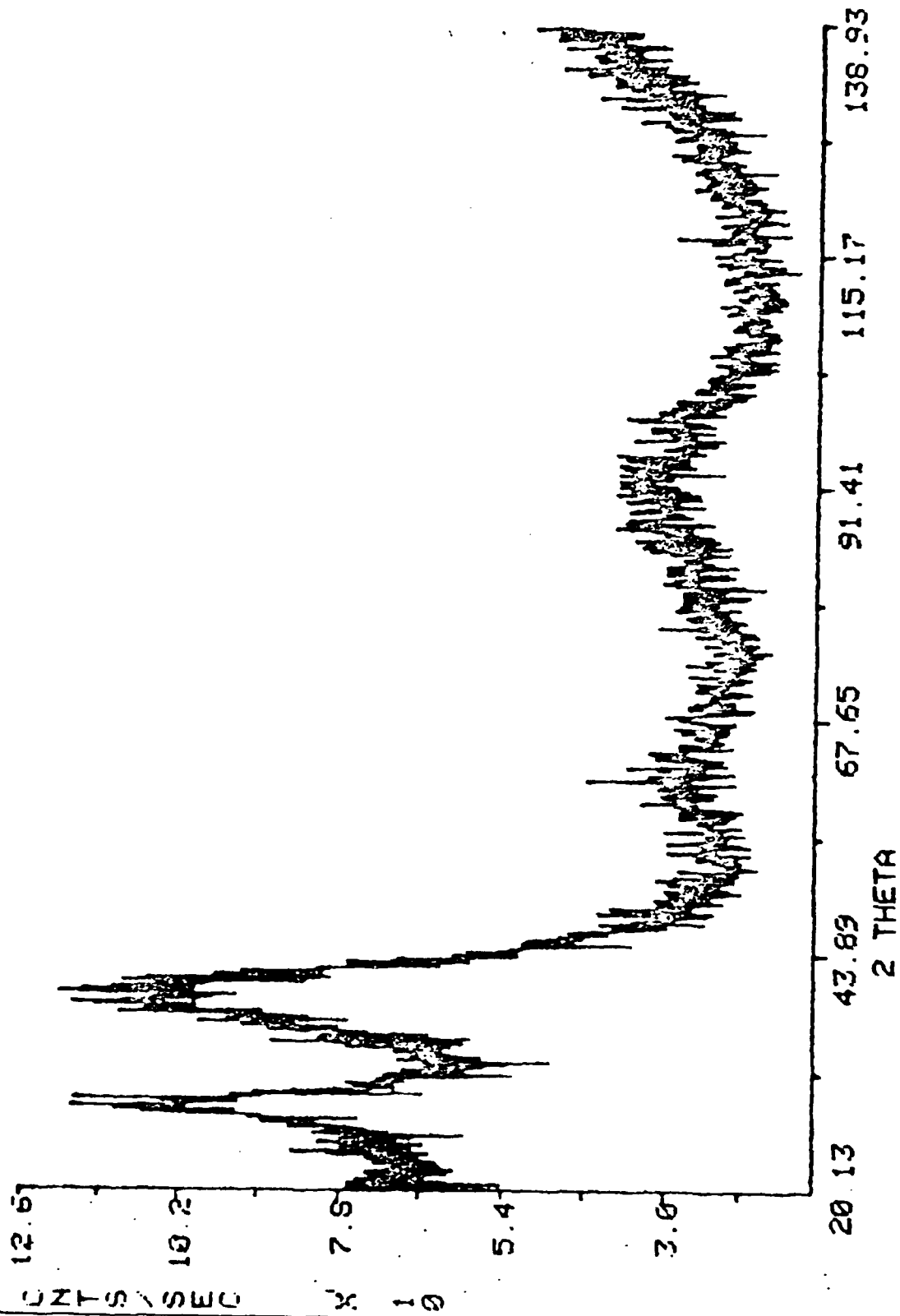
Fig. 5 Sheet resistance of  $2500\text{\AA}$ - and  $3500\text{\AA}$ -thick  $\text{NbSi}_2$  on  $2500\text{\AA}$ -thick  $n^+$  doped poly-Si on  $\text{SiO}_2/\text{Si}$  substrate as a function of annealing time at  $900^\circ\text{C}$  in hydrogen.

Fig. 6 Resistivity of various refractory metal silicides ( $\text{TiSi}_2$ ,  $\text{TaSi}_2$ ,  $\text{MoSi}_2$  and  $\text{NbSi}_2$ ) as a function of annealing temperatures.

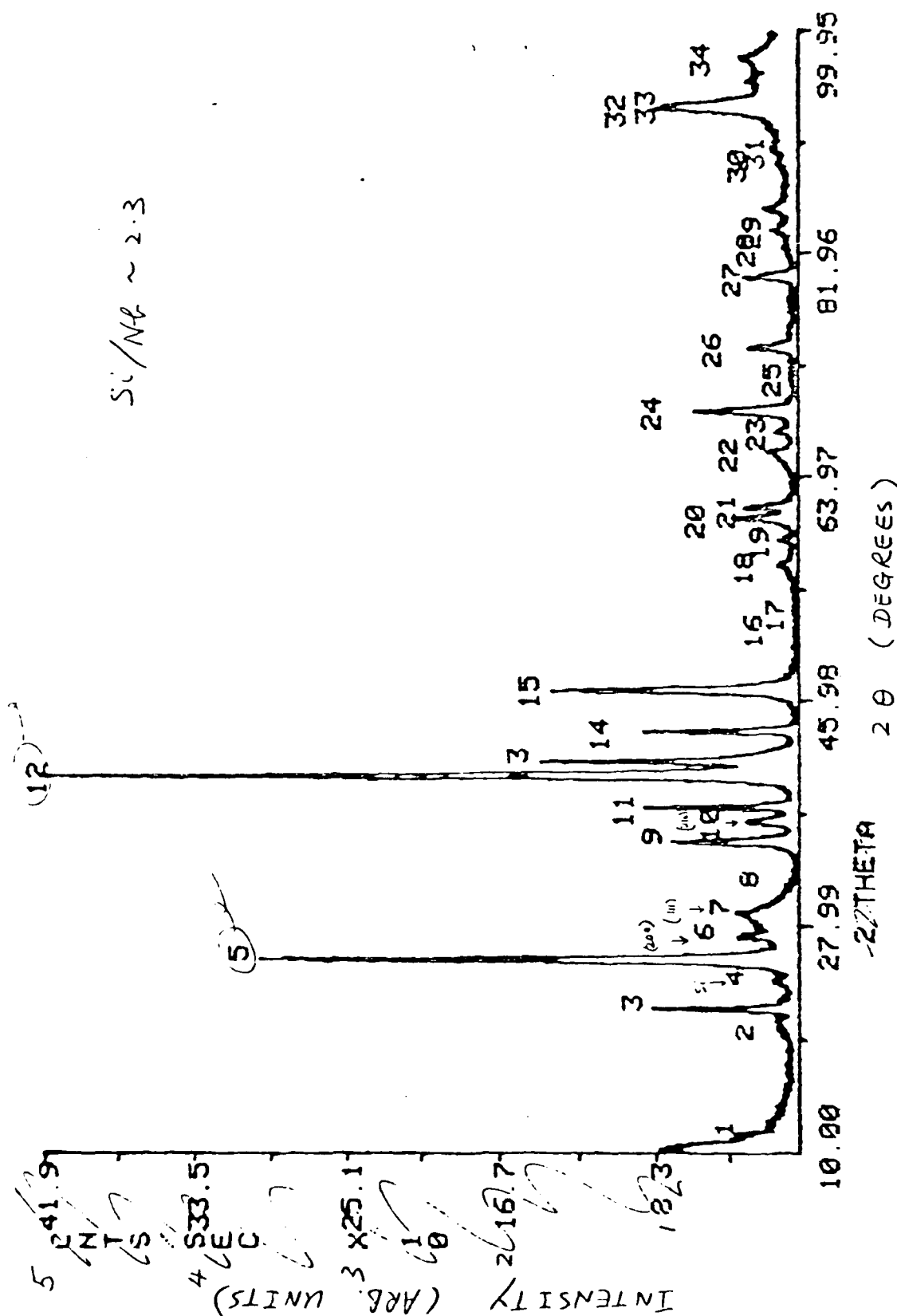
Table I Various silicide phases that are formed for Group IV, V and VIA metals.

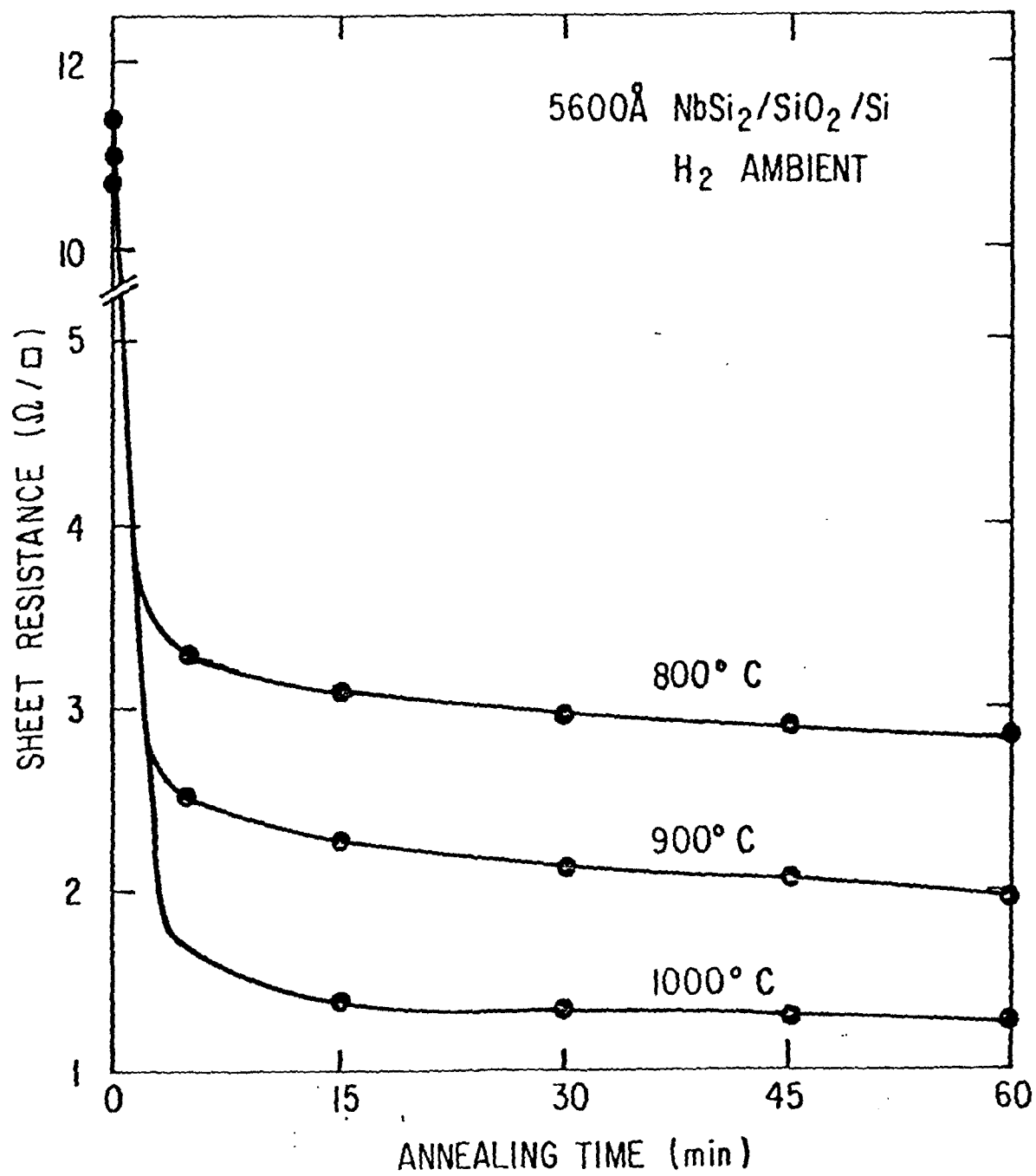
Table II Impurity contents of  $\text{NbSi}_2$  films sputtered from hot-pressed and cold-pressed targets as determined from RBS and SIMS.

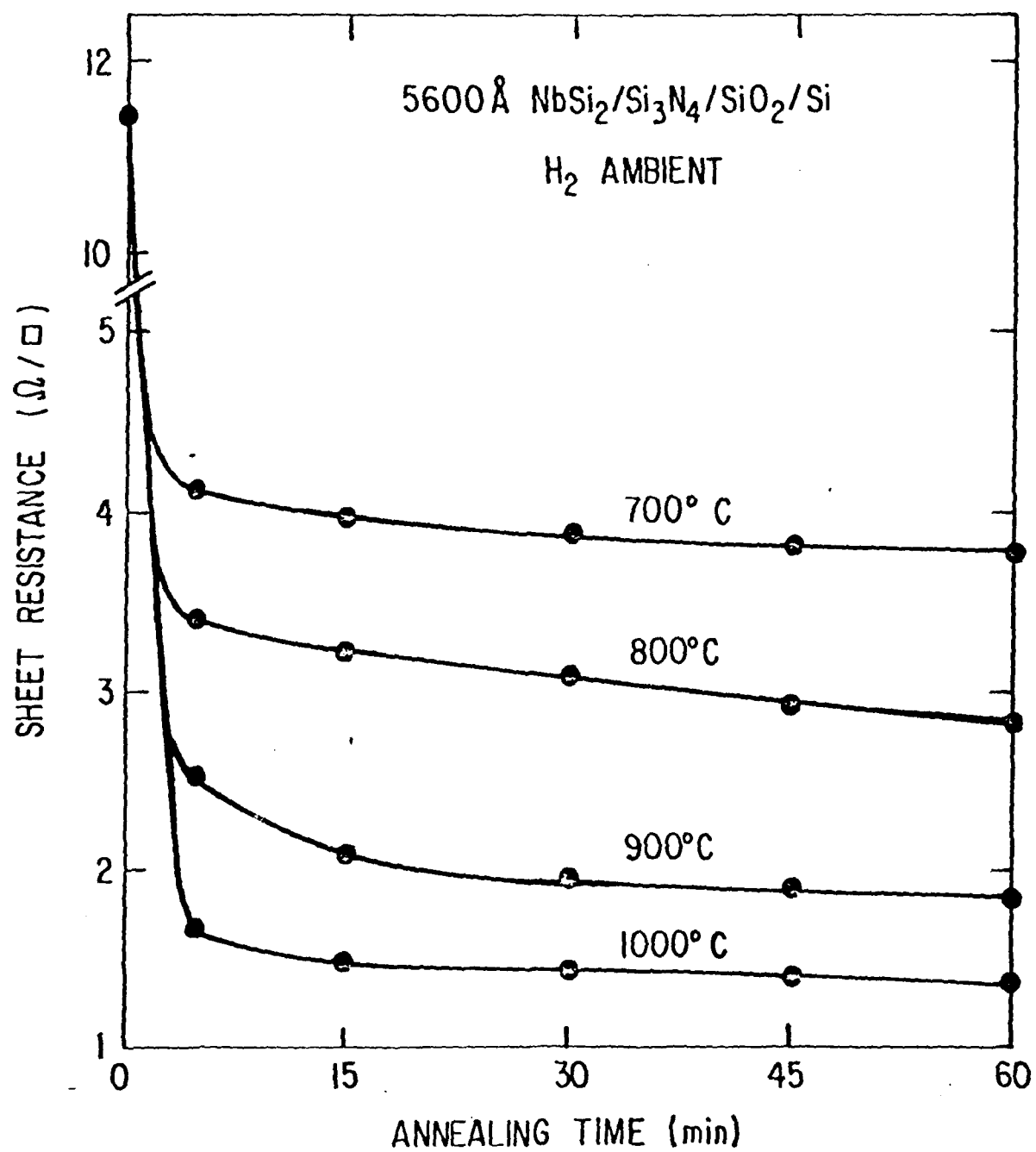
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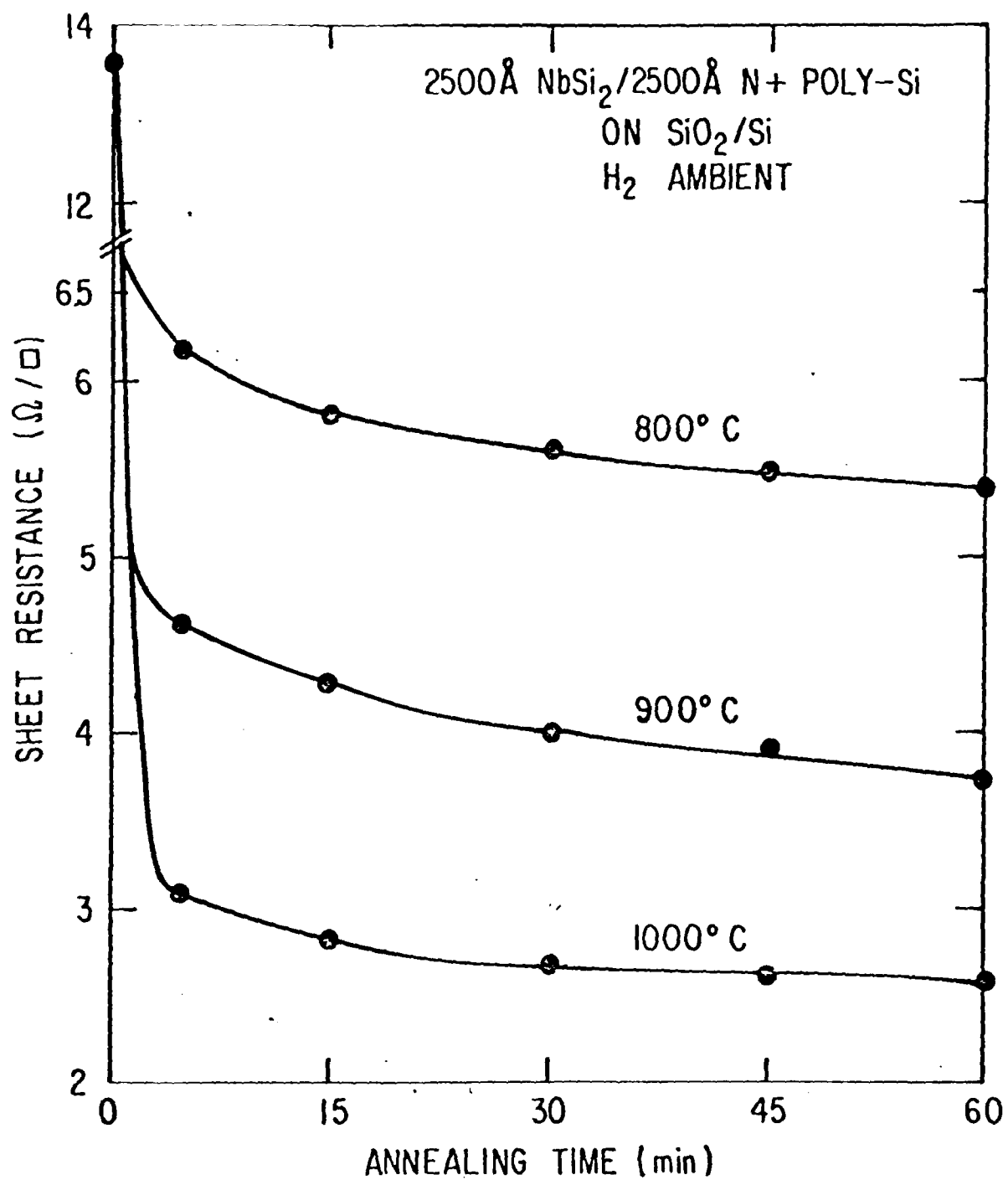
19 5- 5-83 OIF 27451 CHOW NB SI 1X.6 J=1.5  
 INITIAL 2THETA= 10.000 DELTA 2THETA=0.050 TIME INTERVAL= 30.00SEC











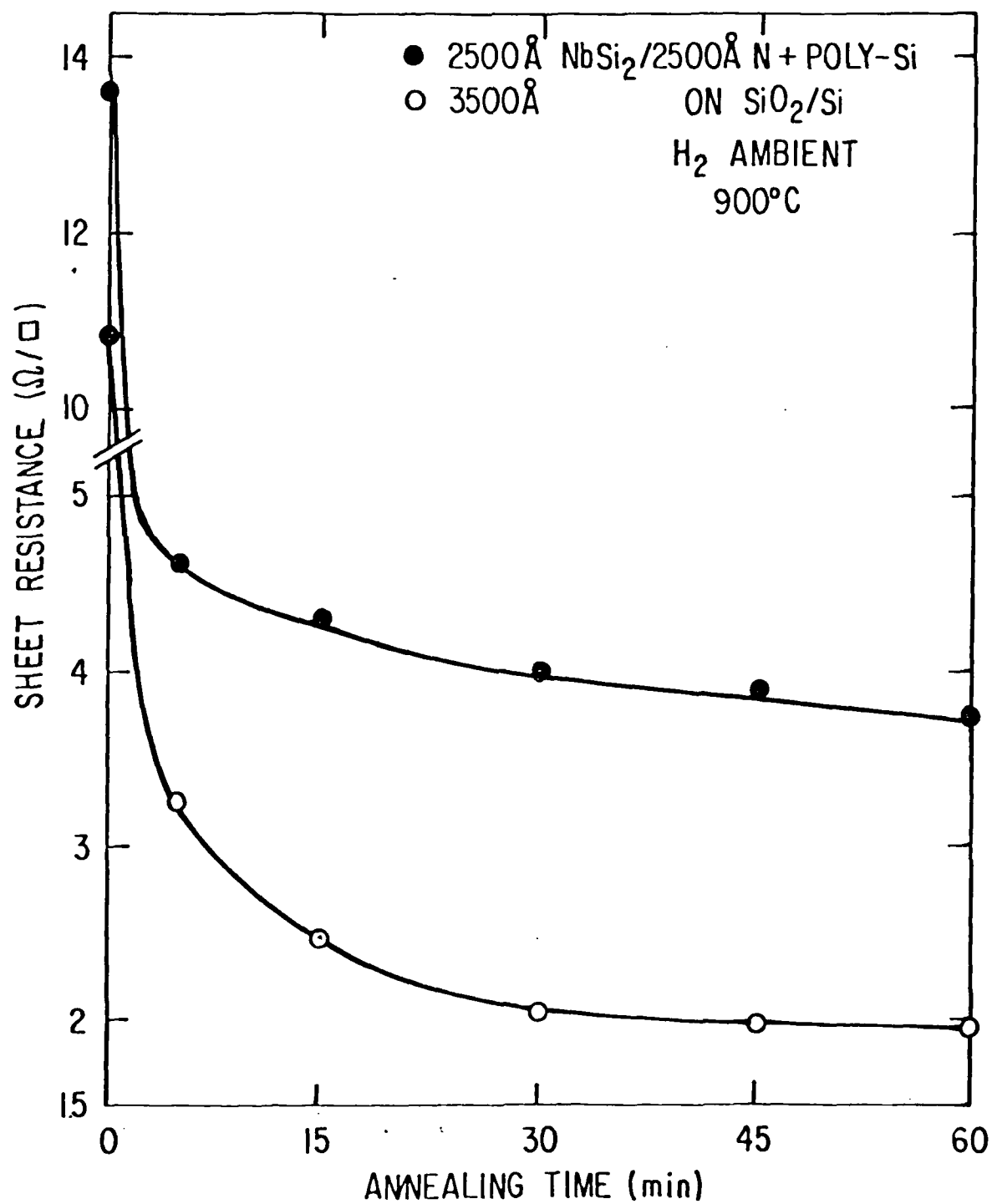


Table I

Various silicide phases that are formed for  
Group IV, V and VIA metals

IV A	V A	VI A
Ti <sub>3</sub> Si	V <sub>3</sub> Si	Cr <sub>3</sub> Si
		Cr <sub>2</sub> Si
Ti <sub>5</sub> Si <sub>3</sub> (C)	V <sub>5</sub> Si <sub>3</sub>	Cr <sub>5</sub> Si <sub>3</sub>
	V <sub>5</sub> Si <sub>3</sub> (C)	Cr <sub>5</sub> Si <sub>3</sub> (C)
		Cr <sub>3</sub> Si <sub>2</sub>
Ti <sub>5</sub> Si <sub>4</sub>	V <sub>5</sub> Si <sub>4</sub>	
	V <sub>6</sub> Si <sub>5</sub>	
TiSi		CrSi
fco-TiSi <sub>2</sub>	VSi <sub>2</sub>	CrSi <sub>2</sub>
bco-TiSi <sub>2</sub>		
Zr <sub>4</sub> Si	Nb <sub>4</sub> Si	
Zr <sub>3</sub> Si	Nb <sub>3</sub> Si	Mo <sub>3</sub> Si
Zr <sub>2</sub> Si		
	α-Nb <sub>5</sub> Si <sub>3</sub> (D8 <sub>1</sub> )	Mo <sub>5</sub> Si <sub>3</sub>
	β-Nb <sub>5</sub> Si <sub>3</sub> (D8 <sub>1</sub> )	
Zr <sub>5</sub> Si <sub>3</sub> (C)	Nb <sub>5</sub> Si <sub>3</sub> (C) <sup>m</sup>	Mo <sub>5</sub> Si <sub>3</sub> (C)
Zr <sub>3</sub> Si <sub>2</sub>		Mo <sub>3</sub> Si <sub>2</sub>
Zr <sub>4</sub> Si <sub>3</sub>		
Zr <sub>5</sub> Si <sub>4</sub>		
Zr <sub>6</sub> Si <sub>5</sub>		
ZrSi		
ZrSi <sub>2</sub>	NbSi <sub>2</sub>	h-MoSi <sub>2</sub>
		t-MoSi <sub>2</sub>
	Ta <sub>4</sub> Si <sub>5</sub>	
	Ta <sub>4</sub> Si	
	Ta <sub>3</sub> Si	W <sub>3</sub> Si
Hf <sub>2</sub> Si	Ta <sub>2</sub> Si	
	Ta <sub>5</sub> Si <sub>3</sub>	W <sub>5</sub> Si <sub>3</sub> (D8 <sub>m</sub> )
	α-Ta <sub>5</sub> Si <sub>3</sub> (D8 <sub>1</sub> )	
	β-Ta <sub>5</sub> Si <sub>3</sub> (D8 <sub>1</sub> )	
Hf <sub>5</sub> Si <sub>3</sub> (C)	Ta <sub>5</sub> Si <sub>3</sub> (C) <sup>m</sup>	W <sub>5</sub> Si <sub>3</sub> (C)
Hf <sub>3</sub> Si <sub>2</sub>		W <sub>3</sub> Si <sub>2</sub>
Hf <sub>4</sub> Si <sub>3</sub>		
Hf <sub>5</sub> Si <sub>4</sub>		
HfSi		
HfSi <sub>2</sub>	TaSi <sub>2</sub>	h-WSi <sub>2</sub>
		t-WSi <sub>2</sub>

Table II

Impurity contents of NbSi<sub>2</sub> films sputtered from  
hot-pressed and cold-pressed targets as determined from  
RBS and SIMS.

	Hot-Pressed	Cold-Pressed	Hot-Pressed/Cold-Pressed
C	1.8	1.0	1.8
O	8	10	0.8
Ar	20	8	2.5
SiN	25	5.5	4.5
(From SIMS)			
Si/Nb	1.7-1.8	2.1	
	2.0(Target)	2.3(Target)	
Ar	3%	3%	
Ta	0.2%	0.2%	
(From RBS)			
Target	99.6%	99.6%	
Purity			

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